The Present and Future Use of High-Energy X-rays for Industrial Materials Research

Yan Gao
GE Global Research Center
Niskayuna, NY 12309

Workshop on "Science with High-Energy X-rays"
August 9, 2004, Advanced Photon Source, ANL

imagination at work

Acknowledgement

Beamlines XOR 1-ID-C, 5-BM-D

Ulrich Lienert (APS)
Jon Almer (APS)
Peter Lee (APS)
Dean Haeffner (APS)
Peter Chupas (ANL)
Qing Ma (DND-CAT)

Beamlines X17B1, X15A

Zhong Zhong (NSLS)

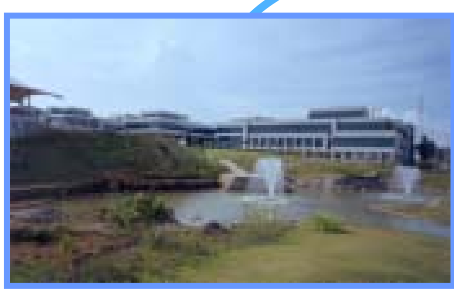
Bill Carter (GE)
Jim Ruud (GE)
Tom Angeliu (GE)
Kan Kump (GEMS)

Outline

- GE and GE Global Research
  - The Present
    - •Residual stress measurement
    - •Characterization of TBC
    - •High throughput XRD and SAXS
    - •Hg XRF at 83 keV
    - •Pr EXAFS at 42 keV
    - •Other applications with HE X-rays
    - The Future
      - •Cutting-edge capability
      - •Advanced characterization
      - •A friendly user facility

GE and GE Global Research

Niskayuna, NY



India

China

Germany

imagination at work

GE Global Research: Hub for innovation

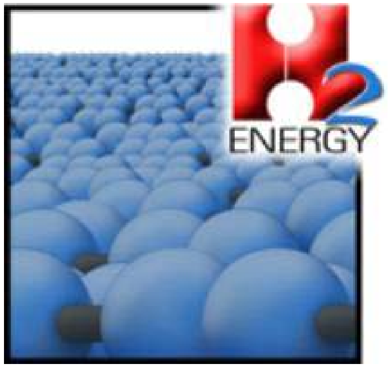
Then…

Discovery of synchrotron radiation at GE Research Center (1947)

And Now…

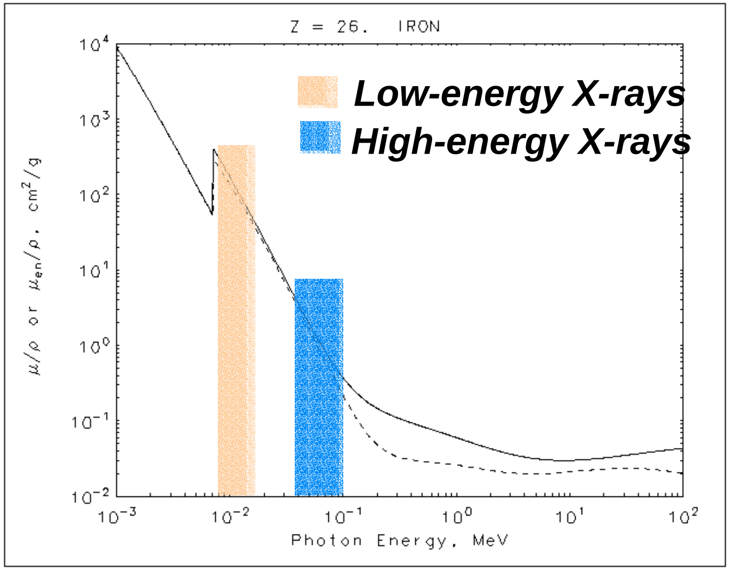
Cutting-edge researc

- *Nanotechnology & functional materials*
- *Hydrogen storage materials*
- *Solid Oxide Fuel Cell*
- *Photovoltaics*



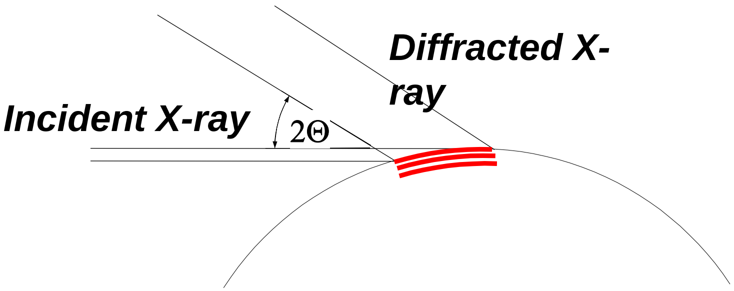
Why is GE interested in HE X-rays

- Unique capability (penetrating power. intensity)
- Superior data quality (S/N, angular resolution)
- Productivity (simpler sample prep, fast data collection)



Research on many metals, alloys, and ceramics

Residual stress measurement



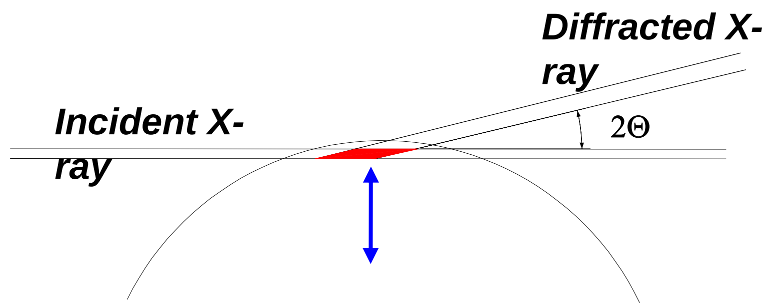
Obtain depth profile by layer removal

In-house X-ray source

- Large beam footprint
- Low intensity for high-angle peaks
- Low accuracy
- Layer-removal for depth profile

Synchrotron X-rays

- Small beam size
- High intensity
- High accuracy
- Non-destructive with HE X-rays



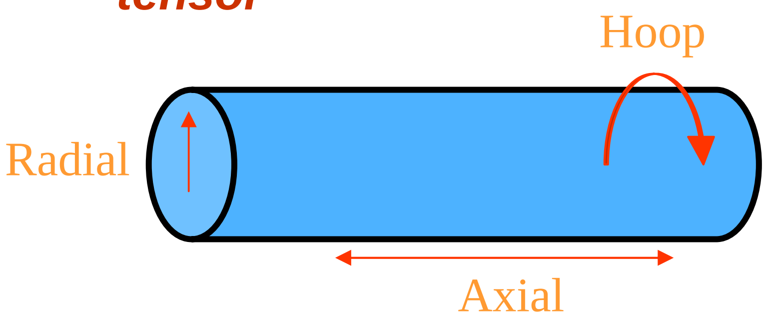
Obtain depth profile by moving sample

Residual Stress Determination Is Very Important for Industry

Non-destructive residual stress measurement

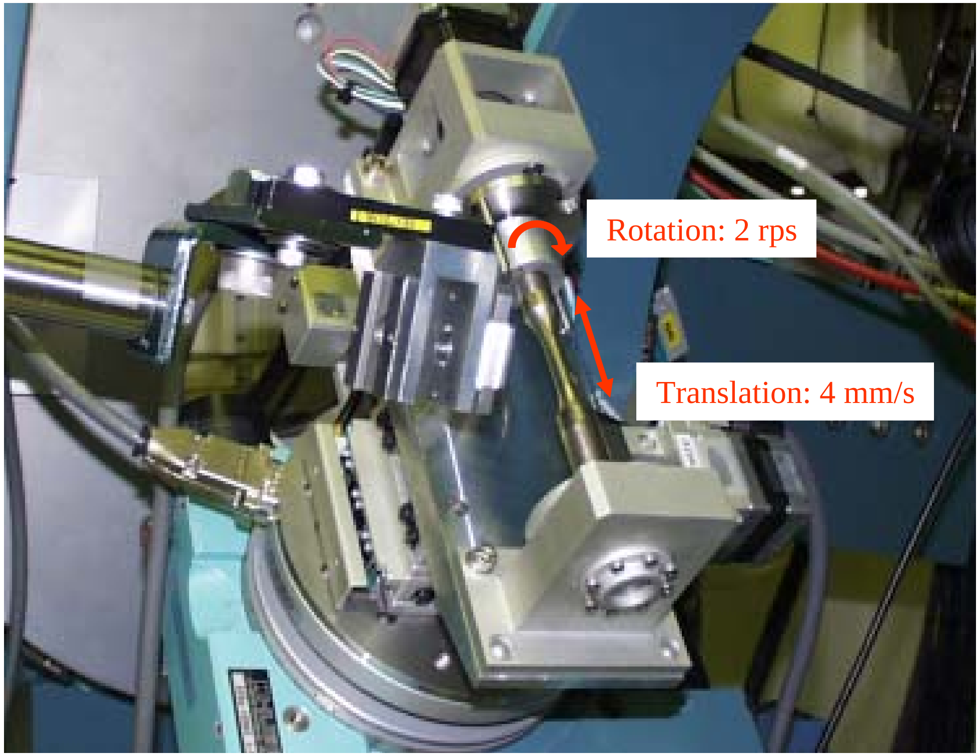
- Shot-peening effect was measured as a <u>function of depth</u>
- Sample stage was used to bring more grains to diffraction

Obtaining triaxial strain tensor



Diffraction data were collected at various χ, ϕ and t (depth up to 1.3 mm)

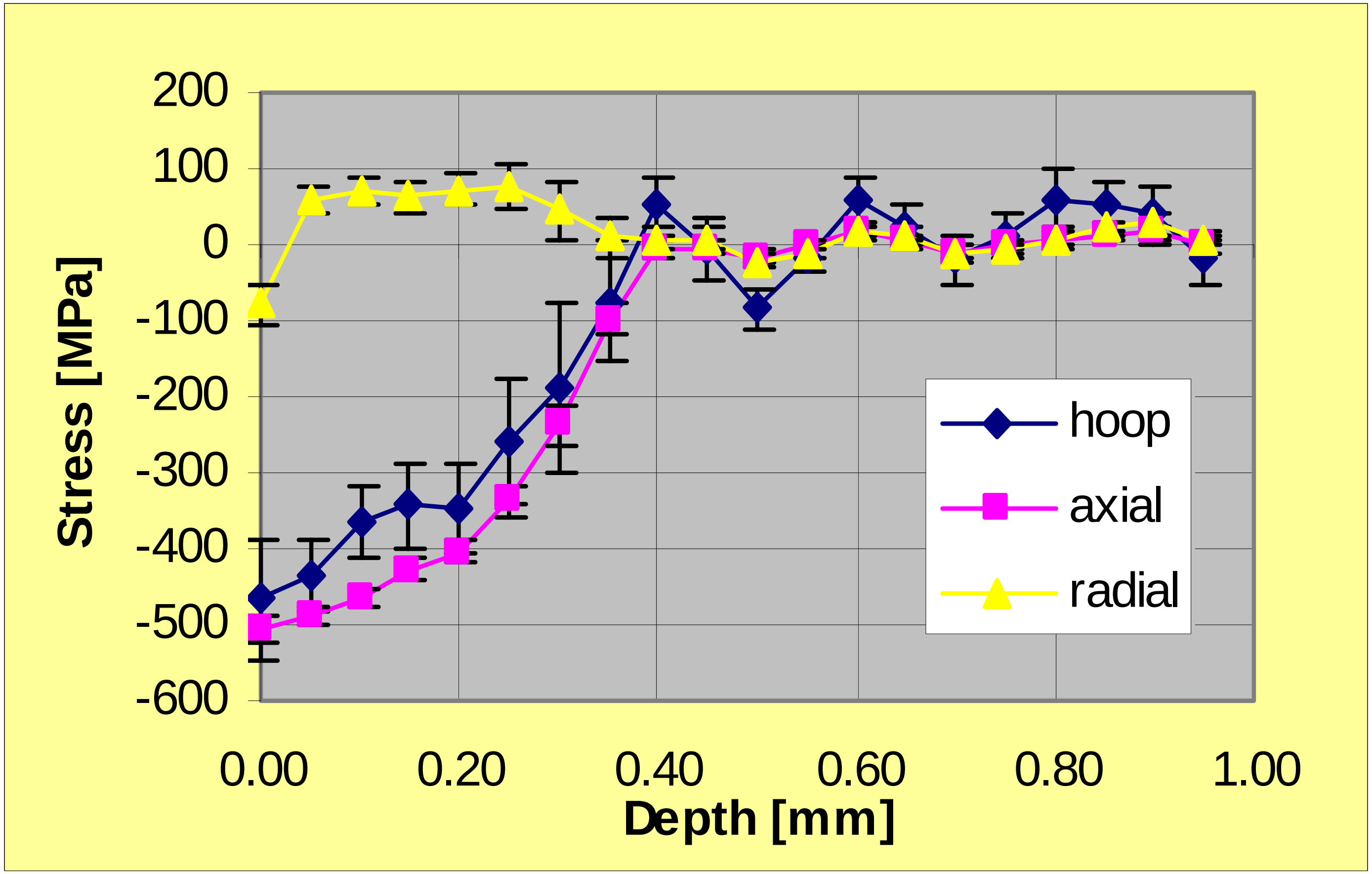
352 images were taken in 6 hours per sample in automated operation



XOR 1-ID-C (U. Lienert)

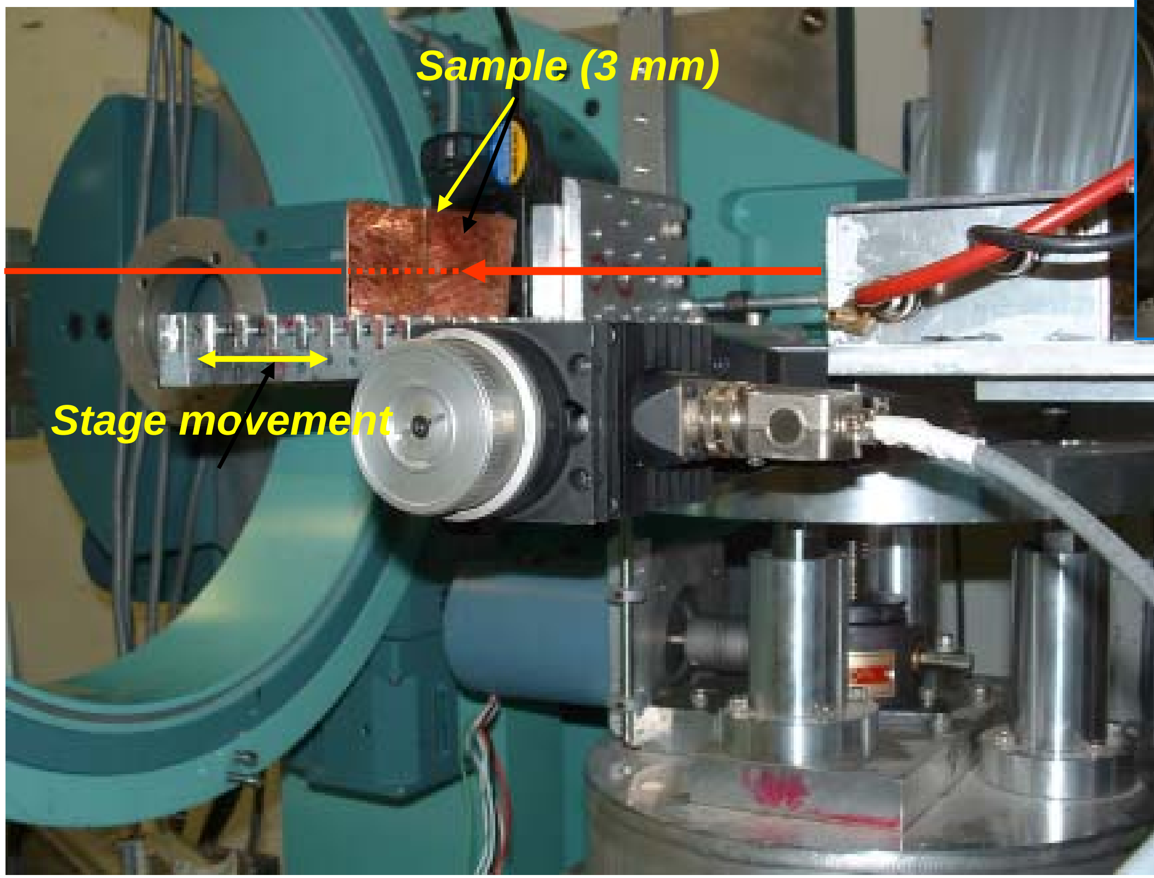
Non-destructive residual stress measurement

- Triaxial stress components determined
- Affected depth up to 400 μm.



High-energy X-ray powder diffraction

Combined with MarCCD and an auto sampler, high throughput measurements can be performed .



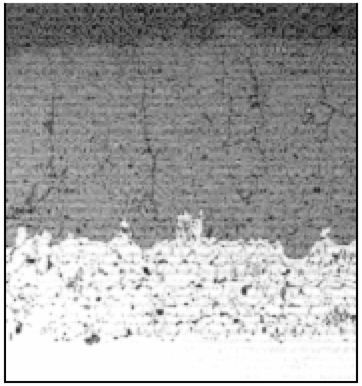
Sagittal focusing monochromator

- *Providing 10^{11} ph/s at 67 keV by focusing horizontal beam from 40 mm to 0.5 mm, which is an flux increase by 2 order of magnitude.*
- *Vertical divergence is between 10-30 micro-radians good angular resolution*

X17B1 (Z. Zhong)

Applications to Thermal Barrier Coatings (TBC)

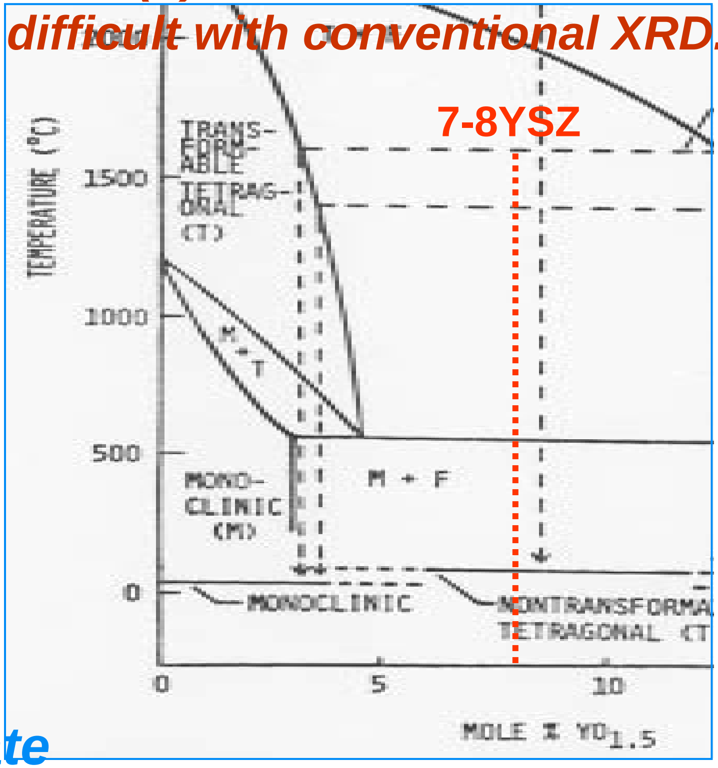
- *Polymorphs: tetragonal, cubic and monoclinic*
- *Separation of tetragonal and cubic peaks*
- *Determination of lattice parameters and c/a': transformable (t) and non-transformable (t')*
- *Texture: difficult with conventional XRD.*



YS Z

Bond coat

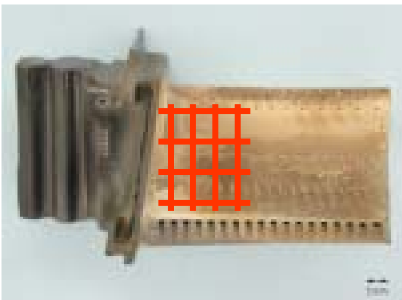
Superalloy substrate



imagination at work

Applications to Thermal Barrier Coatings (TBC)

TBC analyses may involve large number of measurements

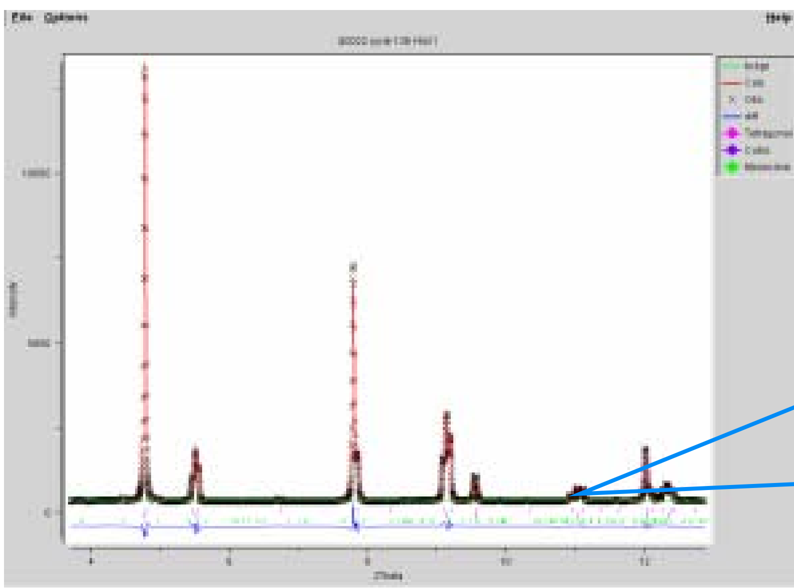


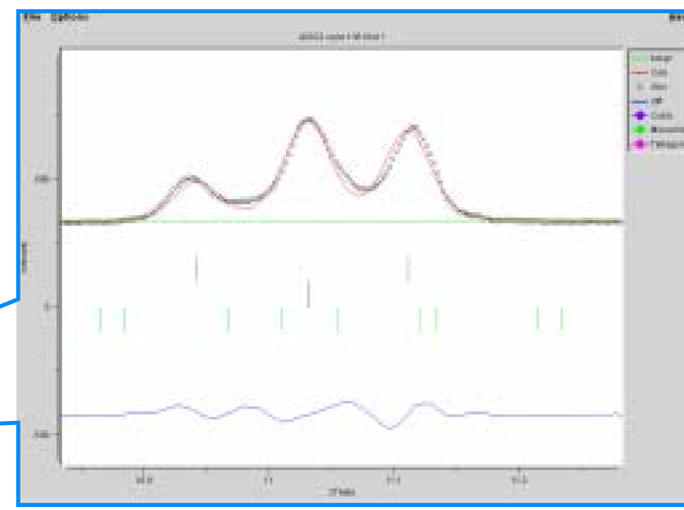
or

Performance DOE

	Temperature		
Time	x	x	x
	x	x	x
	x	x	x
	x	x	x
	x	x	x

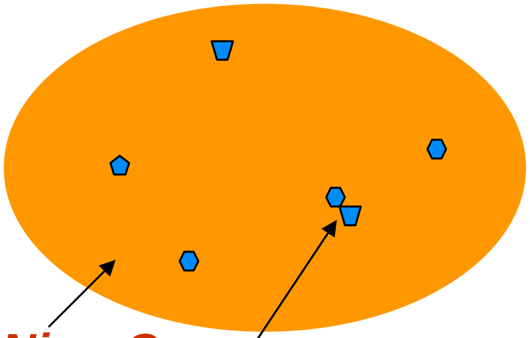
- *Tetra = 49.9 wt%*
- *Cubic = 47.9 wt%*
- *Mono = 2.2 wt%*
- *Tetra c/a' = 1.0154 → t'*
- *Tetra c/a' → 4.4 mol% $YO_{1.5}$*
- *Tetra c/a' → thermal history*
- *Cubic a → 14.0 mol% $YO_{1.5}$*
- *Peak width → micro-strain*
- *Peak position → macro-strain*



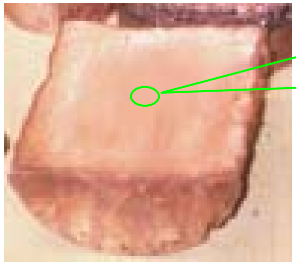


Applications to cast ODS alloys

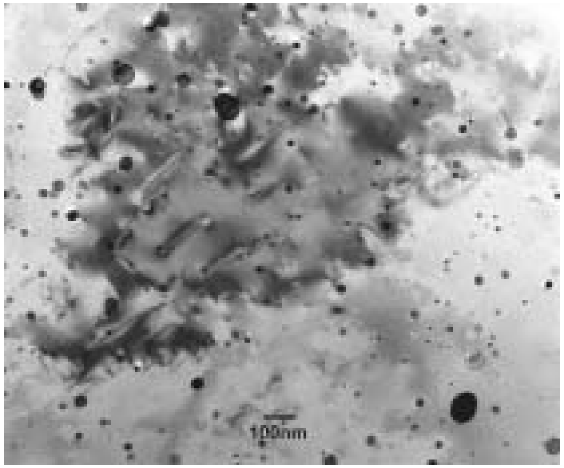
Oxide Dispersion Strengthened A



Conventional analyses



- XRD phase analysis
- TEM sample: from a tiny area
- SAXS sample: thin and small



TEM micrograph

Applications to cast ODS alloys

100 mm

Scan sample

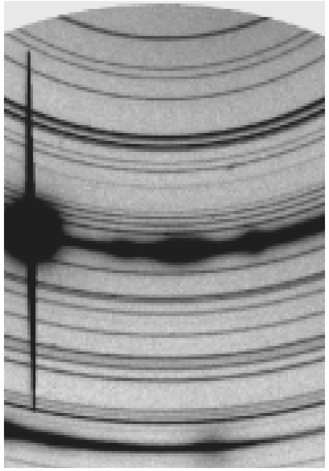
Cross-section of ingot
A few mm thick

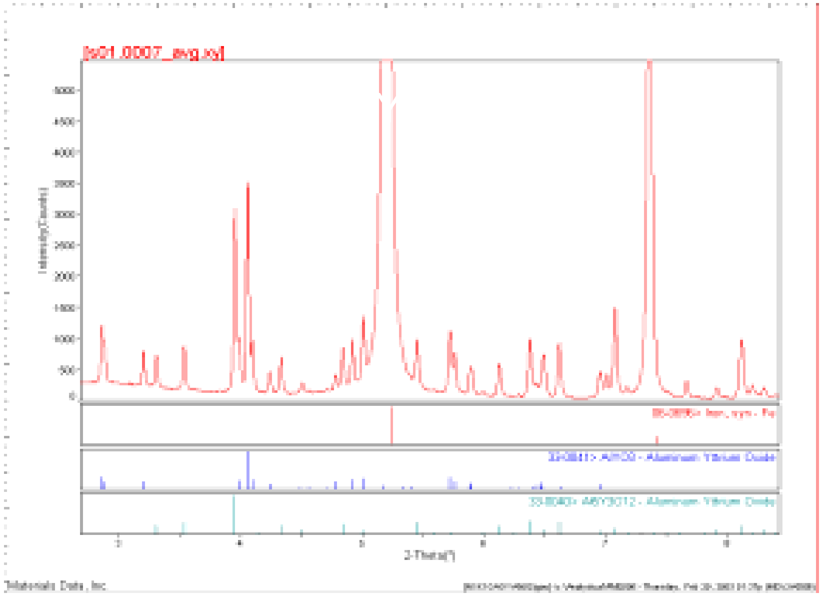
Transmission HE-XRD

- Phase identification
- Oxide dispersion in macro scale

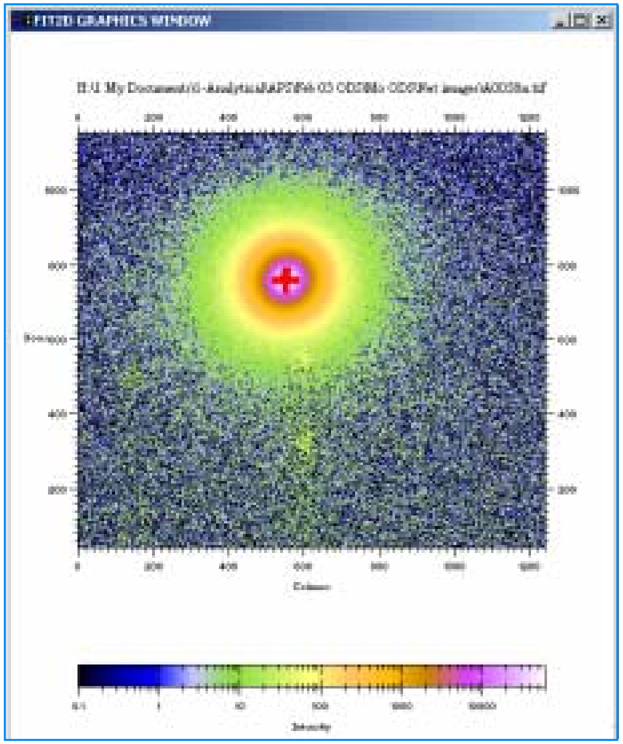
HE-SAXS

- Oxide dispersion in micro scale
- Oxide size and size distribution



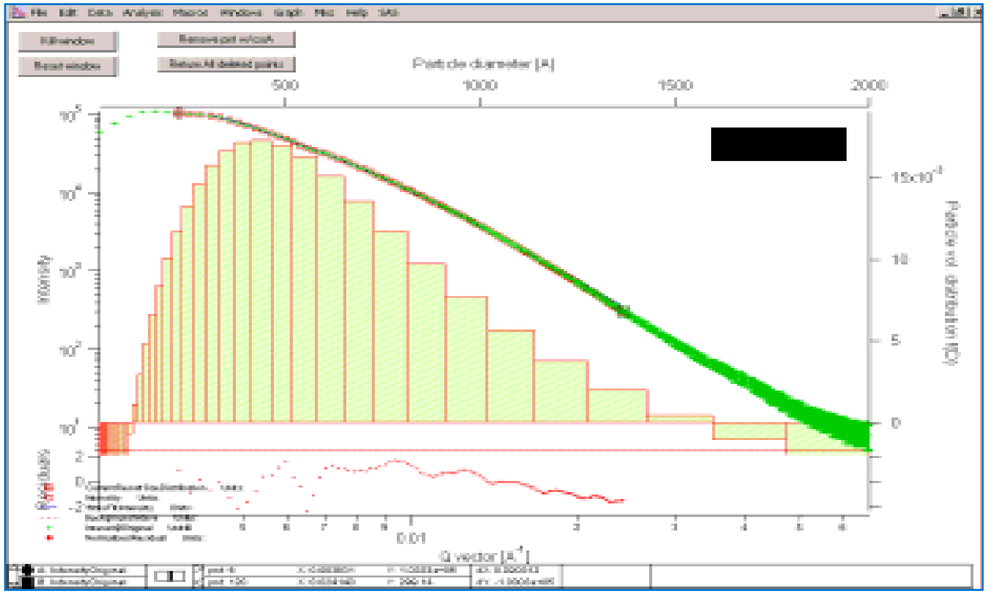


Applications to cast ODS alloys



HE-SAXS

- Oxide dispersion in micro scale
- Oxide size and size distribution



Size and size distribution

Igor + SAS macro (J. Ilavsky)

XOR 1-ID-C (J. Almer)

imagination at work

High-energy X-ray fluorescence

Binding Energy (keV) at K edge

IA	IIA	IIIB	IVB	VB	VIB	VIIB	VIIIB			IB	IIB	IIIA	IVA	VA	VIA	VIIA	0
1 H 1.008																	2 He 4.003
3 Li 6.941	4 Be 9.012											5 B 10.81	6 C 12.01	7 N 14.01	8 O 16.00	9 F 19.00	10 Ne 20.18
11 Na 22.99	12 Mg 24.31											13 Al 26.98	14 Si 28.09	15 P 30.97	16 S 32.06	17 Cl 35.45	18 Ar 39.95
19 K 39.10	20 Ca 40.08	21 Sc 44.96	22 Ti 47.90	23 V 50.94	24 Cr 52.00	25 Mn 54.94	26 Fe 55.85	27 Co 58.93	28 Ni 58.70	29 Cu 63.55	30 Zn 65.38	31 Ga 69.72	32 Ge 72.59	33 As 74.92	34 Se 78.96	35 Br 79.90	36 Kr 83.80
37 Rb 85.47	38 Sr 87.62	39 Y 88.91	40 Zr 91.22	41 Nb 92.91	42 Mo 95.94	43 Tc (98)	44 Ru 101.1	45 Rh 102.9	46 Pd 106.4	47 Ag 107.9	48 Cd 112.4	49 In 114.8	50 Sn 118.7	51 Sb 121.8	52 Te 127.6	53 I 126.9	54 Xe 131.3
55 Cs 132.9	56 Ba 137.3	57 * La 138.9	72 Hf 178.5	73 Ta 180.9	74 W 183.9	75 Re 186.2	76 Os 190.2	77 Ir 192.2	78 Pt 195.1	79 Au 197.0	80 Hg 200.6	81 Tl 204.4	82 Pb 207.2	83 Bi 209.0	84 Po (209)	85 At (210)	86 Rn (222)
87 Fr (223)	88 Ra (226.0)	89 ** Ac (227)	104 Rf	105 Ha	106 Unh	107 Uns	108	109 Une									
			* 58 Ce 140.1	59 Pr 140.9	60 Nd 144.2	61 Pm (145)	62 Sm 150.4	63 Eu 152.0	64 Gd 157.3	65 Tb 158.9	66 Dy 162.5	67 Ho 164.9	68 Er 167.3	69 Tm 168.9	70 Yb 173.0	71 Lu 175.0	
			** 90 Th 232.0	91 Pa (231)	92 U 238.0	93 Np (244)	94 Pu (242)	95 Am (243)	96 Cm (247)	97 Bk (247)	98 Cf (251)	99 Es (252)	100 Fm (257)	101 Md (258)	102 No (259)	103 Lr (260)	

36.0

90.5

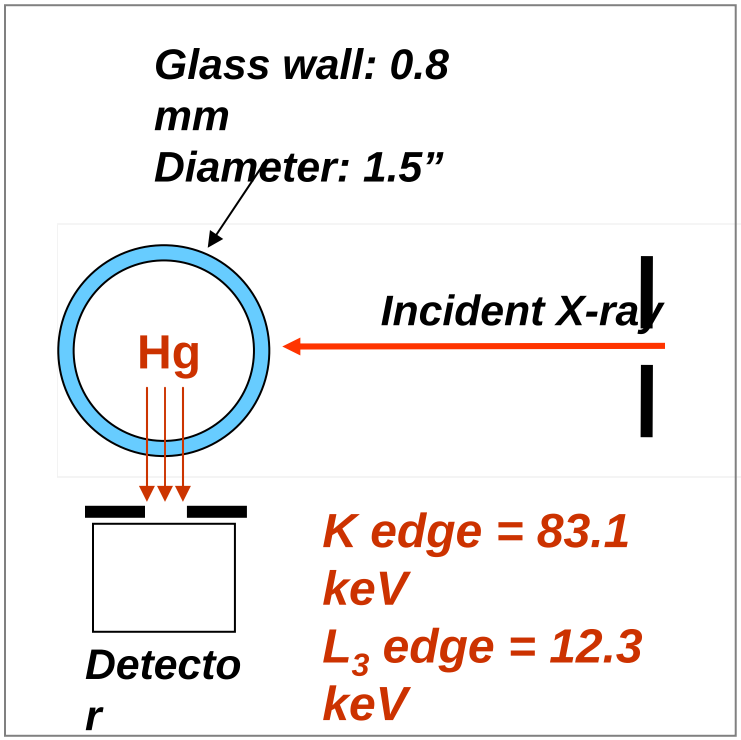
- *Many commonly used elements, including RE elements*
- *Excitation beyond the energy of in-house XRF*
- *Greater fluorescence yield at K-edge*
- *Useful for non-destructive detection*

Example: Hg K-edge

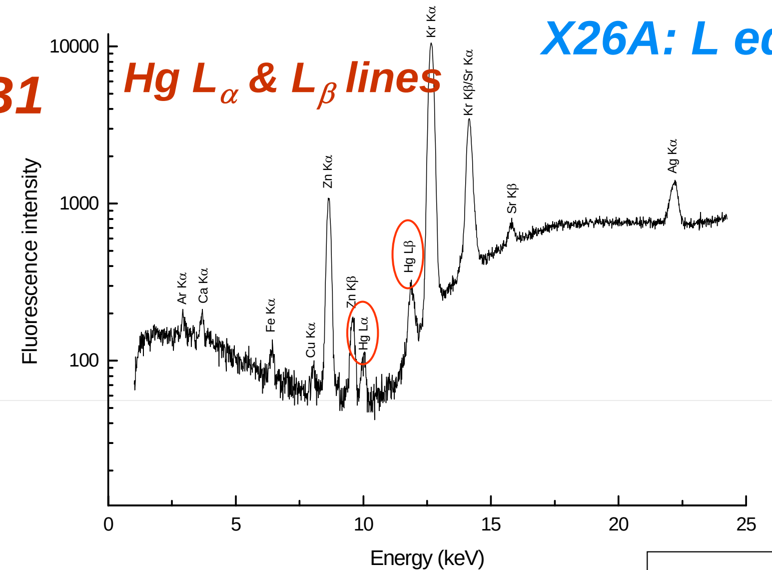
- *Low Hg concentration (a few mg)*
- *Non-destructive*
- *Hg vapor pressure vs. temperature*

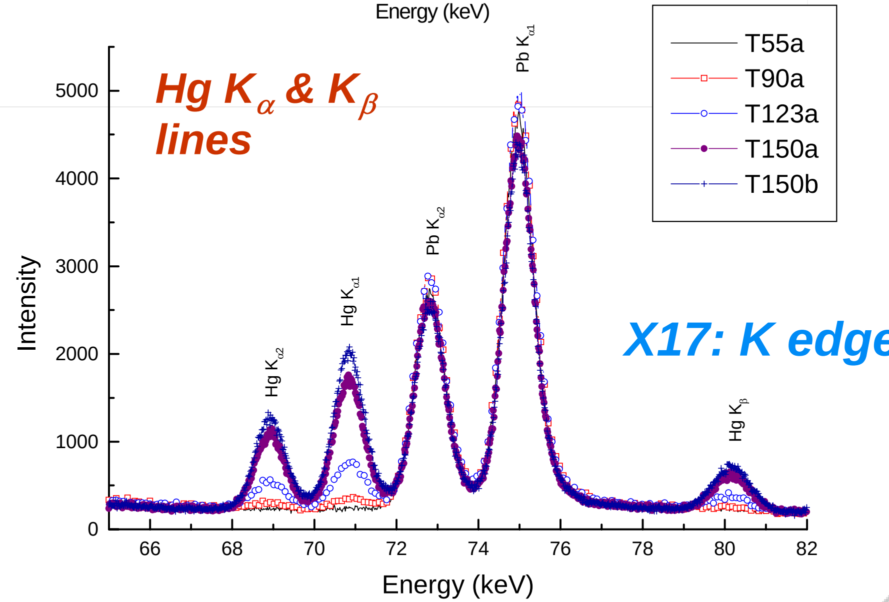
Non-destructive detection of Hg vapor in F-lamps

Setup at NSLS X26A and X17B1



X26A: L edge



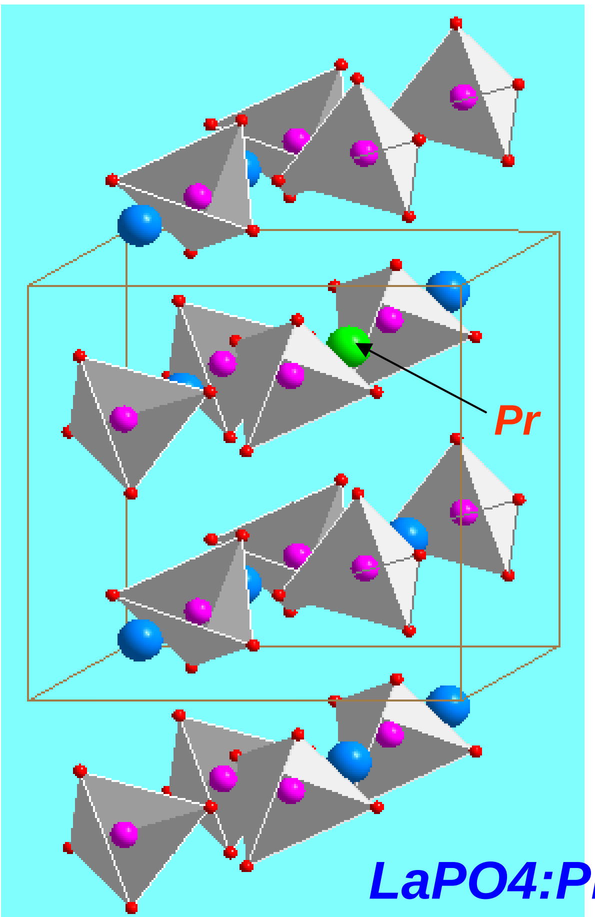


The lamp was wrapped with heating tape for temperature-dependent measurement

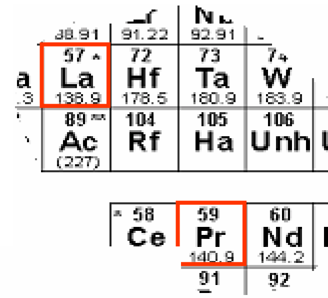
imagination at work

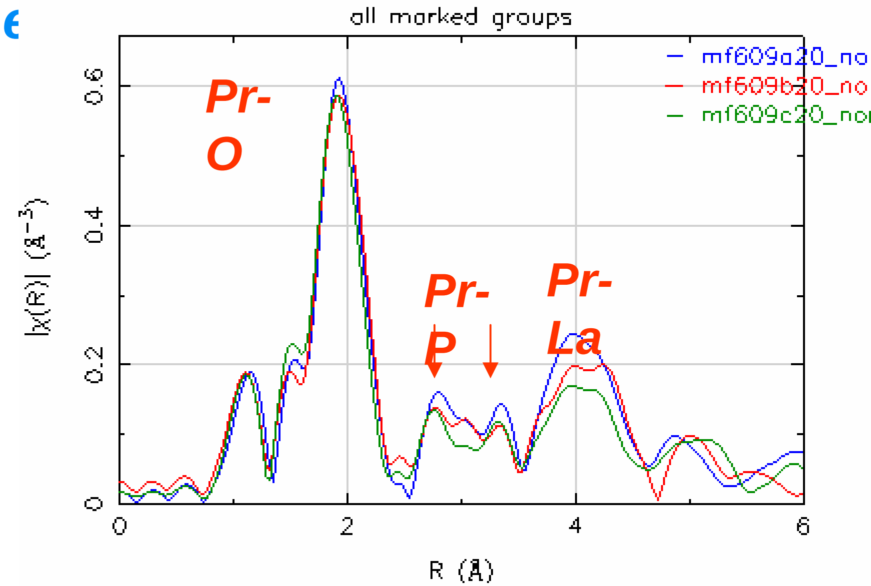
X-ray absorption spectroscopy using HE X-rays

Motivation: Understand the role of Pr doping in Quantum Splitting Ph



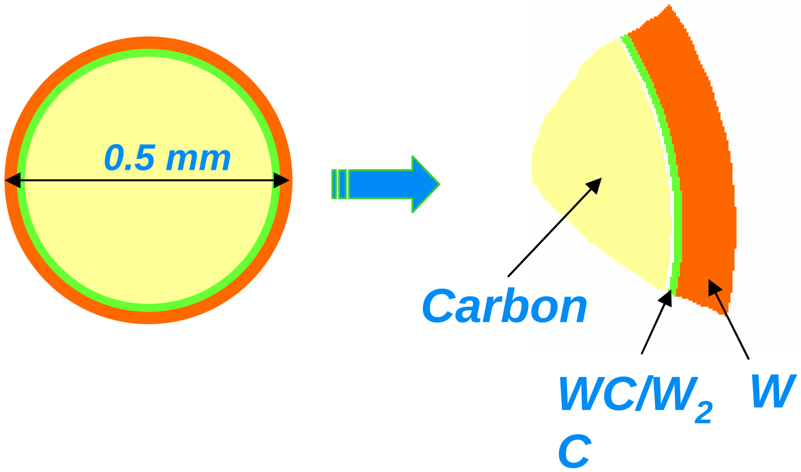
- *Pr L-edge not possible due to La*
- *Pr K-edge not possible at*





K-edge data from APS 5-BM-D

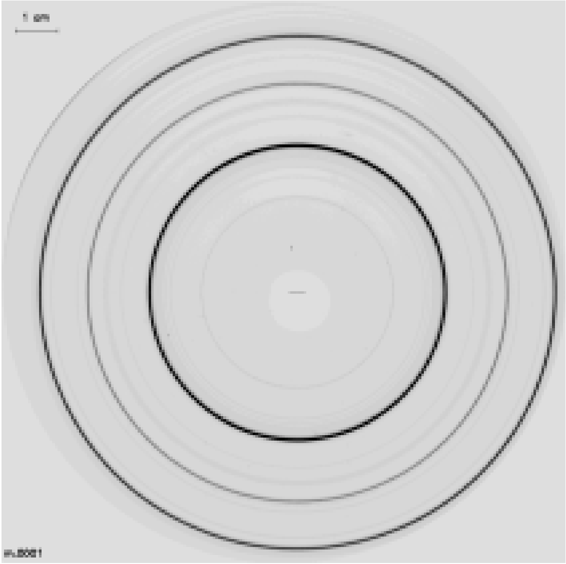
Quantitative phase analysis using HE X-rays

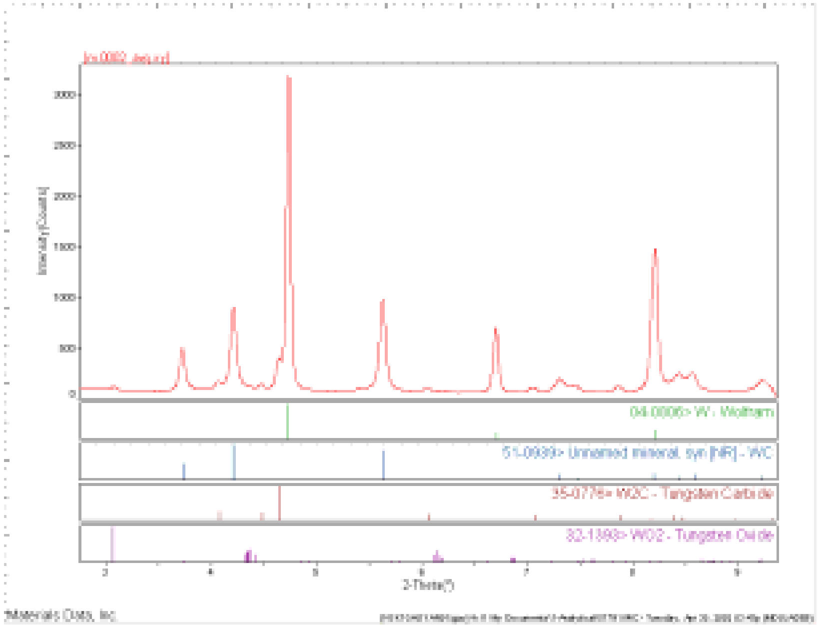


<u>Objective</u>: Quantify W, WC and W_2C

Tungsten absorption is too severe for in-house conventional X-rays

<u>Solution</u>: High-energy XRD at 67 keV!





imagination at work

Non-destructive XRD using HE X-rays


QuEnergy

Patents

United States Patent 6,132,823
Qu October 17, 2000

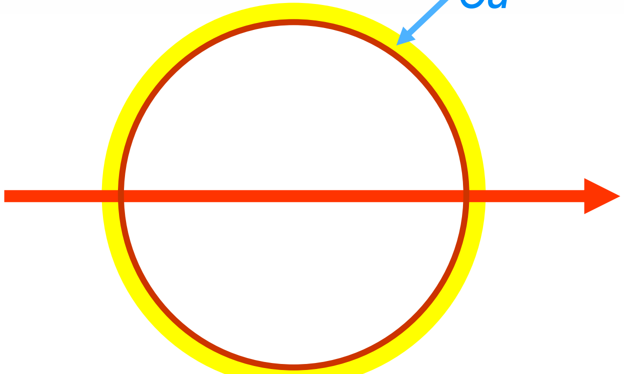
Superconducting heat transfer medium

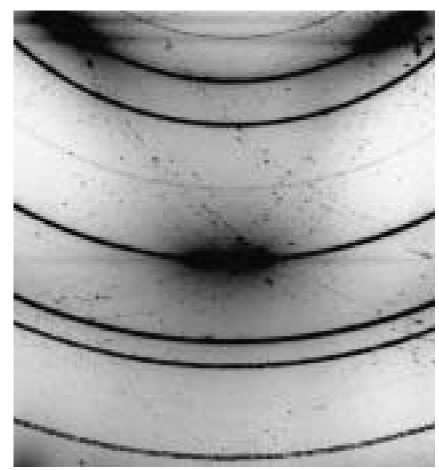
Abstract

A superconducting heat transfer medium that has three basic layers, the first layer being various combinations of sodium, beryllium, a metal such as manganese or aluminum, calcium, boron and dichromate radical; the second layer formed over the first layer and being various combin
cobalt, manganese, beryllium, strontium, rhodium, copper, .beta.-titanium
calcium, a metal such as manganese or aluminum and the dichromate
former

How does it work and why?

The pipe is claimed to have several layers, and work only when it is sealed, therefore HE-XRD is the chosen technique to investigate the interior chemistry and crystal structure.





67 KeV X-ray in transmission mode

The Future

Cutting-edge capability

- *A turnkey facility for MicroXRD and microXRF*
- *Fast time-resolved in-situ diffraction*

Advanced characterization

- *Non-destructive residual stress*
- *High-throughput materials screening (XRD, XRF and SAXS)*

A friendly user (including industrial users) facility

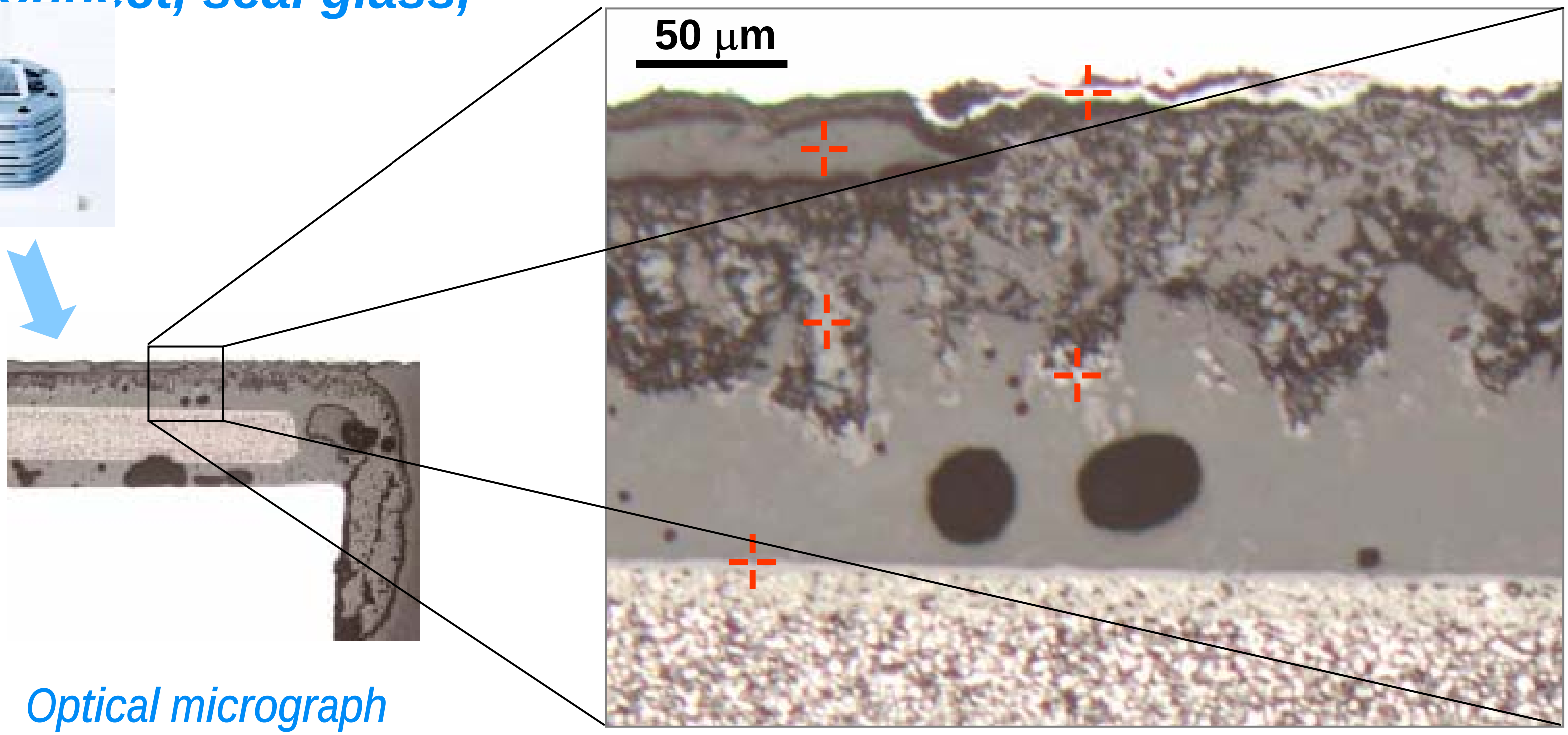
- *Dedicated instrumentation for frequently used techniques*
- *Quick access and/or remote access*
- *Commercialized analytical services*

Microdiffraction with high spatial resolution

Cross-section of a SOFC part:

Consisting cathode, anode, electrolytes, interconnect, seal glass,

While elemental information may be obtained by SEM-EDS, it's very important to obtain <u>crystal structure</u> information from region of interest as marked.



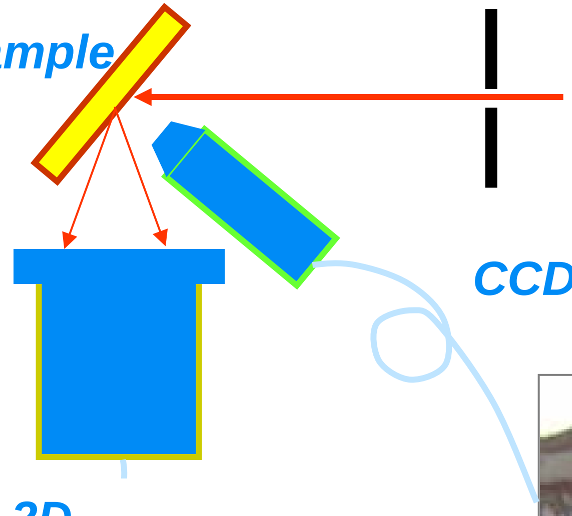
Optical micrograph

A turnkey microdiffraction station: aim and shoot

A wavelength dispersive <u>XRF</u> detector can be added

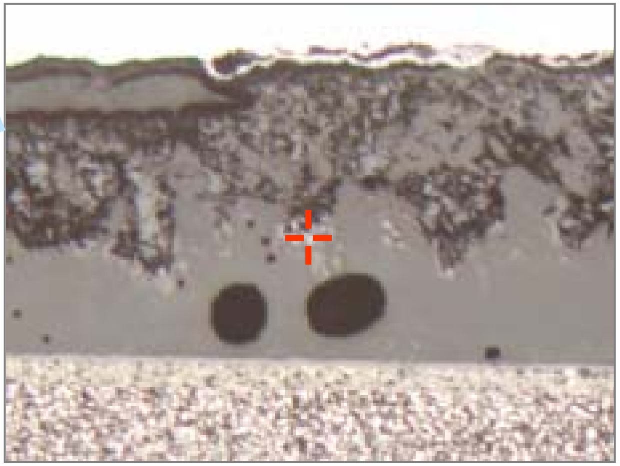
Monochromatic beam for polycrystalline area

Sample

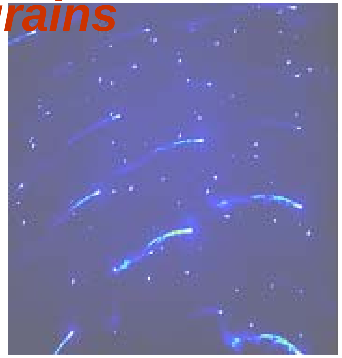


Interchangeable, focused white and monochromatic beam of a few μm across

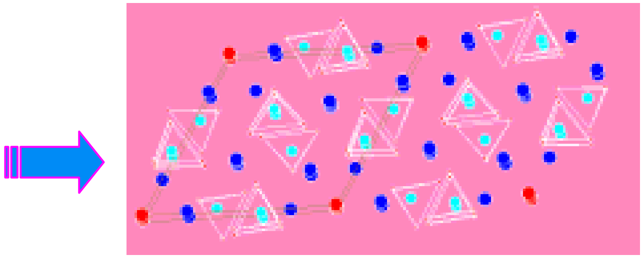
CCD Camera



White beam for single crystal grains



2D detector



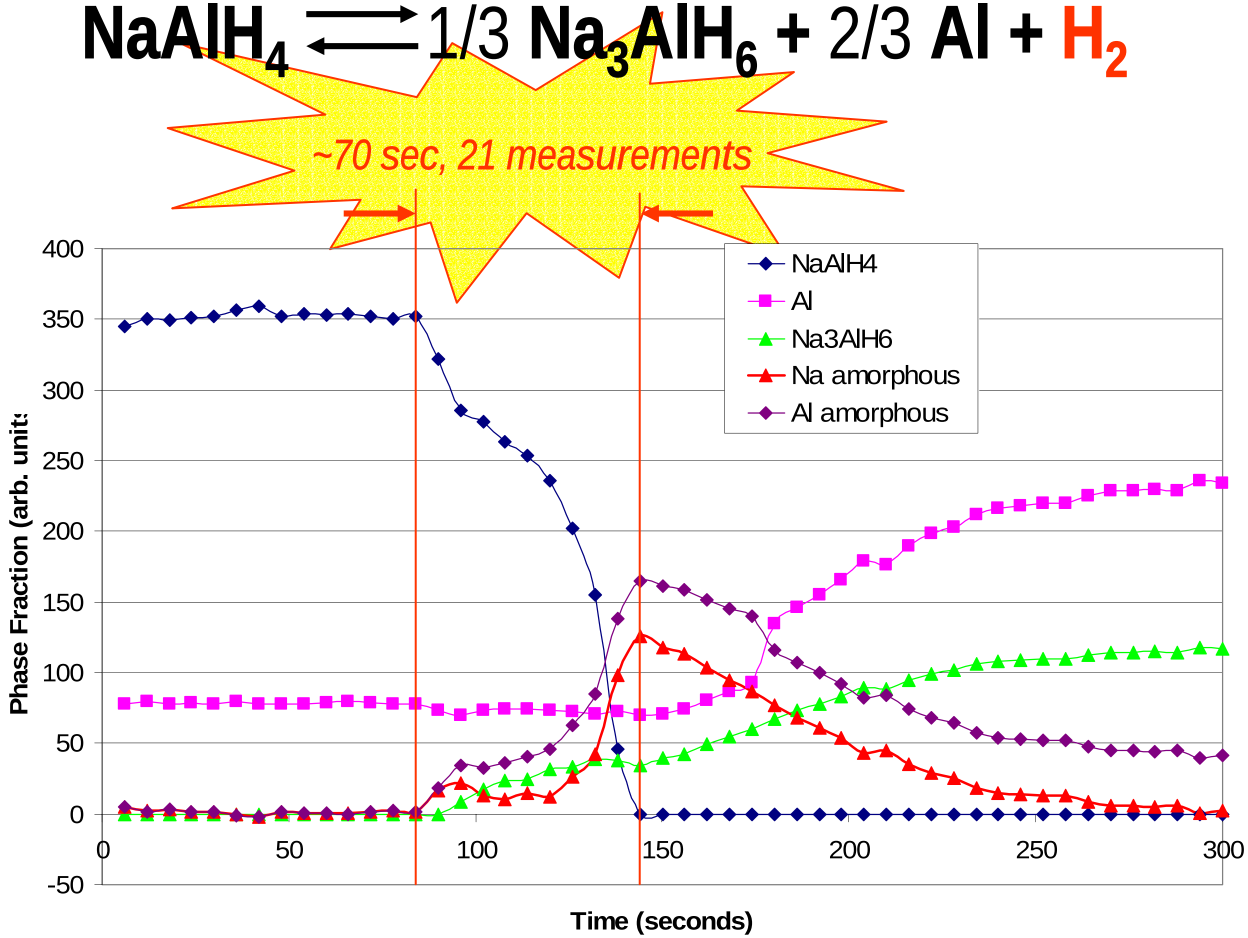
Crystal structure

imagination at work

Fast time-resolved XRD

Time resolution can be essential for mechanistic understanding!

$$NaAlH_4 \rightleftharpoons 1/3\ Na_3AlH_6 + 2/3\ Al + H_2$$

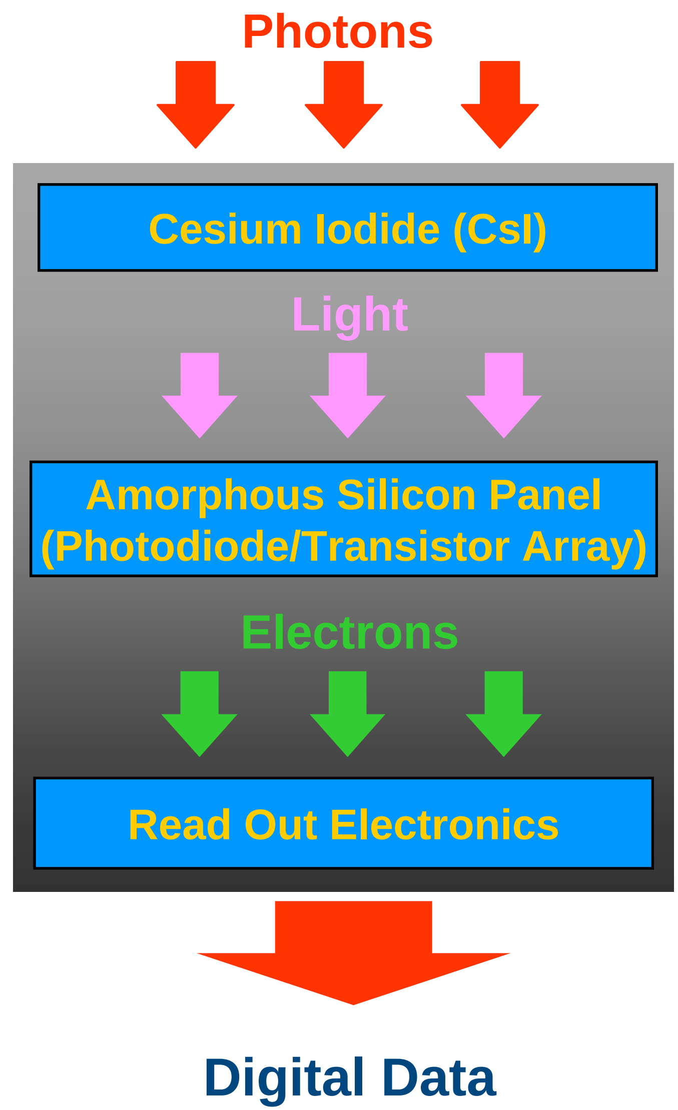


XOR 1-ID-C using GE 2D detector (P. Chupas and P. Lee)

Fast time-resolved XRD with GE Detector

GE detector at work at XOR 1-ID-C

- *Area: 41 cm x 41 cm*
- *Pixel size: 200 μm*
- *Readout: 41 ms for 2k x 2k (Angio)*
- *Dynamic range: 14 bits*

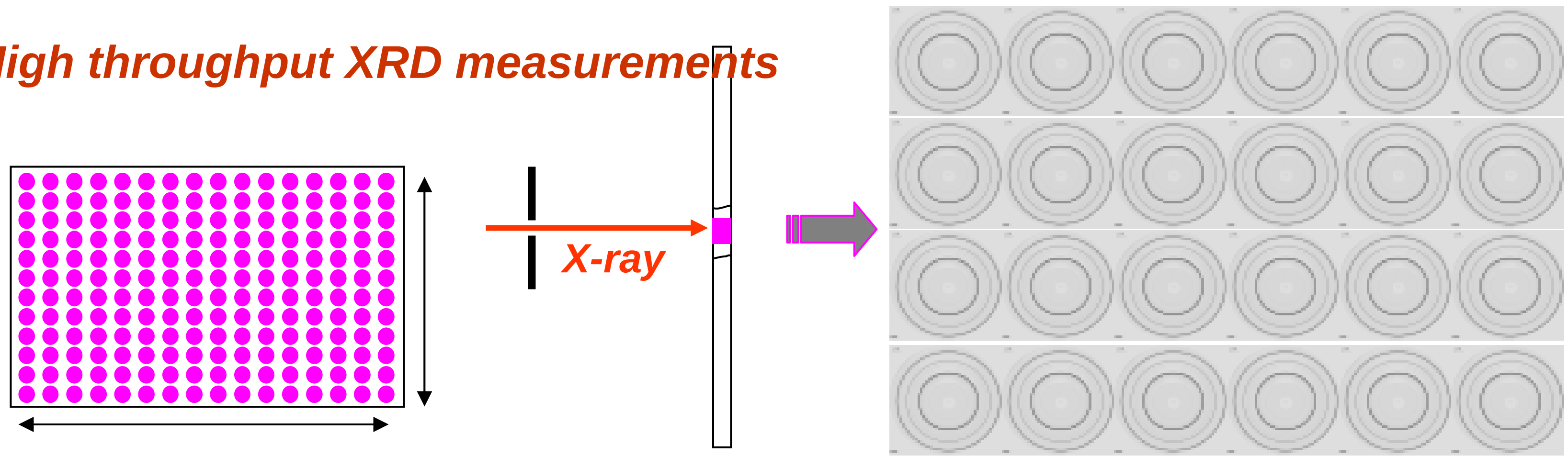


Contact: Ken Kump, GE Medical Systems, Ph: 262-548-4549

imagination at work

Advanced Characterization

High throughput XRD measurements



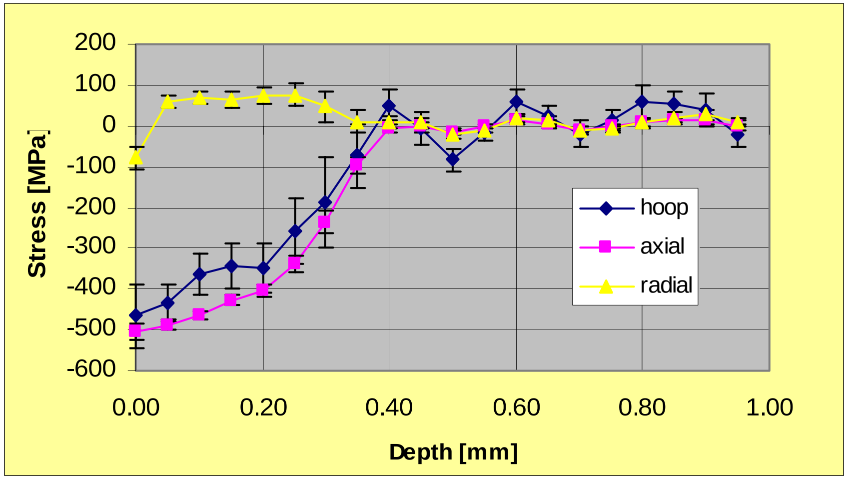
With automated sample stage, 2D detector, high-energy X-rays, superb synchrotron intensity, and dedicated data analysis software, large number of samples can be preloaded and measured unattended or remotely. High-energy X-rays in transmission mode is particularly useful for many inorganic or metallic materials.

Same approach, combined with micro-focused beam, can also be used for <u>automated diffraction mapping</u> with monochromatic beam, or <u>elemental mapping</u> with white beam.

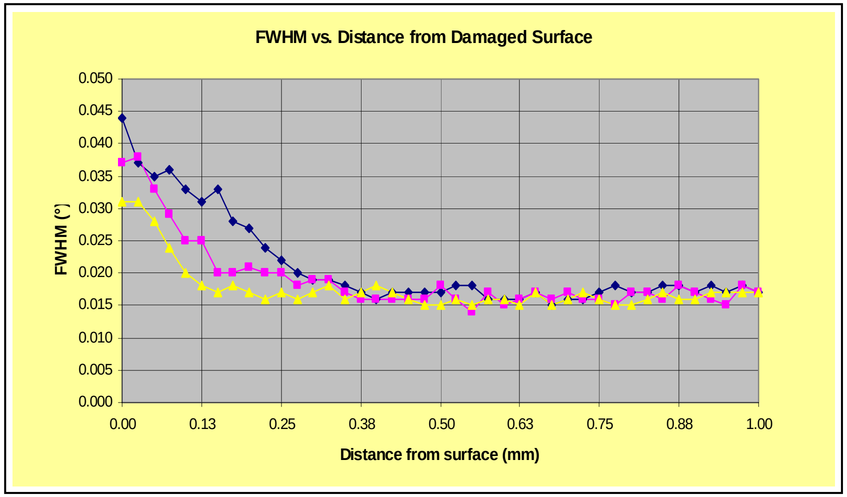
Advanced Characterization

- *Residual stress and plastic deformation are very important for industrial applications*
- *Actual samples involves complex geometry, and small beam and high intensity are essential for obtaining accurate data*
- *Non-destructive with HE X-rays*
- *Consider dedicated instrumentation and commercialize the analyses through a third party.*

Residual stress



Plastic deformation



imagination at work

The Future

A friendly user (including industrial users) facility

- **Dedicated instrumentation for frequently used techniques**

    Such as powder diffraction with 2D detector for normal or high throughput applications

- **Quick access and/or remote access**

    Linked with dedicated instrument to minimize setup time; web-based remote access for users running experiment from home institution

- **Commercialized analytical services**

    Powder diffraction and residual stress measurement may be two key areas to promote fee-based analytical services